

## REPORT ON MAPLE PRODUCTS.

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Three samples of maple sirup and three of maple sugar were sent out. Sugar No. 4 was made in the laboratory from Sirup No. 1; Sugar No. 5, from Sirup No. 2; and Sugar No. 6, from Sirup No. 3. Dry basis results from the sugars should be comparable with those from the corresponding sirups.

### PREPARATION OF THE SAMPLE.

The directions to collaborators called for a study of the desirability of adding water to all samples, whether sirup, sugar or other maple products, boiling to 104°C. (219°F.) and filtering after such concentration was completed. A comparison of the merits of filter paper, muslin, and cotton wool as filtering media was also requested.

The reboiling and filtering of sirups before analysis was recommended by C. H. Jones in 1905<sup>1</sup> and suggested to the association at its twenty-second annual convention<sup>2</sup>. The suggestion was endorsed by Bryan at the twenty-ninth convention<sup>3</sup>. Its whole-hearted adoption would simplify the directions materially.

S. F. Sherwood favors filtering after the product has been evaporated to the proper density and regards filter paper as a satisfactory medium. The experience of the writer is that filtration of the concentrated sirup through paper is too slow, since the sirup becomes more concentrated during the filtration. Muslin is rather an indefinite and perhaps too coarse a medium. The associate referee prefers cotton wool (absorbent cotton) loosely packed in the point of a funnel. This is also open to the charge of indefiniteness but the writer has found it satisfactory.

In one series of experiments, where different portions of the same hot sirup were poured upon filters of the three classes, 100 cc. ran through the muslin, and 50 cc. through the cotton wool, in 2 minutes, while it

<sup>1</sup> Vt. Agr. Expt. Sta. Rept., 1905, 327.

<sup>2</sup> U. S. Bur. Chem. Bull. 99: (1906), 45.

<sup>3</sup> *Ibid.*, 162: (1913), 59.

are required  
 paper. In still  
 through the three media

40 cc.  
 25 cc.  
 paper (single)..... 5 cc.

every instance the portions of sirup filtered through the paper gave a higher refractometer reading than either of the other portions.

The few comparative experiments made upon the analytical values of samples filtered through the different media revealed no material differences. Thus, in Sirups Nos. 1 and 2, results for total ash (dry basis) are as shown in Tables 1 and 2.

and Canadian lead number,  
 respectively,

TABLE 1.  
 Determination of total ash (dry basis) in Sirup No. 1.

ANALYST	COTTON WOOL	FILTER 1 S. & S. NO. 597
	per cent	per cent
G. J. Van Zoeren, Macdonald College, Quebec, Canada . . .	0.88	0.88*
	0.88	0.88*
	0.85	0.85*
	0.81	0.85*
Average.....	0.86	0.87
J. F. Snell.....	0.83	0.81†
	0.92	0.83†
	0.90	....
	0.84	....
	0.88	....
Average.....	0.87	0.82

\* Single.  
 † Double.

TABLE 2. ✓ Canadian Lead Number  
 Determination of total ash (dry basis) in Sirup No. 2.  
 (Analyst, J. F. Snell.)

MUSLIN	COTTON WOOL	FILTER PAPER S. & S. NO. 597 (DOUBLE)
per cent	per cent	per cent
3.48	3.94	3.54
3.56	3.59	3.48
....	3.73	....
....	3.60	....
....	3.50	....
Average.... 3.52	3.67	3.51

The amount of work done upon this point is not sufficient to justify any recommendation other than a continuation of study.

### MOISTURE BY REFRACTOMETER.

TABLE 3.  
*Determination of moisture by refractometer<sup>1</sup>.*

SIRUP NUMBER	ANALYST	
	Van Zoeren	Snell
	<i>per cent</i>	<i>per cent</i>
1.....	32.63	32.66
2.....	34.96	34.96
3.....	32.66	32.70

These determinations were made with the one instrument, a Féry refractometer made by Adam Hilger, London, England.

Results by one observer on ten samples using the Abbé and the Féry instruments were reported by Snell and Scott<sup>2</sup> in 1914 and showed close agreement.

Sherwood is of the opinion that the refractometer method is quite accurate in the case of maple sirup. A. H. Bryan<sup>3</sup> also pointed out the advantages of this method of estimating dry substance in maple sirup as well as in most other liquid saccharine products. In twelve out of thirteen samples he obtained higher results for moisture by the refractometer method than by drying 3 to 5 grams on 10 to 15 grams of sand in a t-bottomed dish at 70°C. in a vacuum oven until the loss in 5 hours did not exceed 3 mg.

### MOISTURE BY DRYING.

The method of drying on sand in a vacuum oven at 70°C., prescribed for honey and tentatively adopted for all maple products, is open to criticism on the grounds of indefiniteness as to the measure of the vacuum, the times for weighing and the degree of constancy to be attained; and the inappropriateness for maple products, which ordinarily contain but little levulose. The Laboratory of the Canadian Department of Inland Revenue follows the practice of drying at 100°C. to constant weight, having the sugar finely powdered and spread upon a watch glass, the sirup on asbestos fiber or sand. The two tentative methods of the association<sup>4</sup> for massecuites, molasses and other liquid and semi-liquid products, *i. e.*,

<sup>1</sup> *Assoc. Official Agr. Chemists, Methods*, 1916, 126.

<sup>2</sup> *J. Ind. Eng. Chem.*, 1914, 6: 216.

<sup>3</sup> *J. Am. Chem. Soc.*, 1908, 30: 1443.

<sup>4</sup> *Assoc. Official Agr. Chemists, Methods*, 1916, 121.

drying upon pumice at 70°C. (in the absence of levulose, 100°C.), and drying upon quartz sand at 100°C., may possibly be suitable for maple products.

None of these methods is regarded as quite satisfactory. With saccharine products, absolute constancy of weight is rarely, if ever, attainable by any method of oven drying and in none of the methods referred to are the conditions so closely defined as to yield concordant results in the hands of different analysts. In the case of sirups, the refractometer method forms a satisfactory substitute, not only giving much more accordant results, but also effecting great economy of time and labor. From the work of West<sup>1</sup> on sorghum sirup, it would appear that Danne's calcium carbide method possesses similar advantages. Both of these methods, however, require special apparatus, and the recognition of a direct drying method is perhaps unavoidable as a concession to the laboratory of modest resources. Such a method must, however, be much more closely defined than any of those at present in use. Provision must be made not merely for uniform temperature throughout the oven, but for uniform ventilation as well. In the determination of moisture in evaporated apples, J. A. Dawson (Laboratory of the Inland Revenue Department, Vancouver, B. C.) reports results differing by one to two units of percentage, depending upon whether the dish stood at the front or back of the shelf of an electrically heated oven of the Freas type. He also states that, in such an oven set for 100°C., he has observed variations from 97 to 103°C. in a calibrated thermometer laid horizontally on the shelf with its bulb in the back left corner. Such observations are suggestive of the need for further improvement of the apparatus available for the determination of moisture by loss of weight by drying.

The points indicated for study under this head were:

- (1) Comparison of drying at 70°C. in a vacuum oven and at 100°C. in a water-jacketed or electrically heated oven.
- (2) Quantity of sample to be used.
- (3) Spreading material—sand, pumice or asbestos—and in the case of sugars, omission of spreading material.

#### EXPERIMENTS AT 100°C.

Dawson made preliminary experiments upon a standard 65 per cent by weight solution of cane sugar, prepared by shaking together at 30°C., 70 grams of water and 130 grams of commercial extra fine granulated pure cane sugar, previously ground to pass a 40-mesh sieve, and dried for 16 hours at 100 to 110°C. The sugar used gave, after the drying, a polarimeter reading of 99.8° Ventzke. The solution obtained

<sup>1</sup> *J. Ind. Eng. Chem.*, 1916, 8: 31.

had a refractive index of 1.4509 at 28°C., as measured by the Abbé refractometer. By Geerlig's tables such a solution contains 64.52 per cent of sugar. The results obtained by drying 2 grams of this solution, plus 10 cc. of water, in a glass crystallizing dish 5.5 cm. in diameter and 3 cm. high in a Freas electric oven, set for 100°C., for exactly 4 hours, were as follows:

TABLE 4.

*Moisture determined by different methods.*

(Analyst, J. A. Dawson.)

SAMPLE NUMBER	SPREADING MATERIAL OMITTED	SAMPLE PLUS 15 GRAMS ACID-WASHED IGNITED SAND	SAMPLE PLUS 5 GRAMS IGNITED CHRYSOTILE ASBESTOS
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
1.....	30.84	34.32	36.19
2.....	30.80	34.24	(35.46)
3.....	29.86	34.42	36.10
4.....	30.53	34.33	35.92

The sand used was fine enough to pass a 20-mesh, but not a 40-mesh sieve. It was stirred at the beginning of the experiment and after 1 and 2 hours.

In the case of asbestos No. 2, it was observed that the spreading material was not in contact with more than half of the bottom of the dish and evidently some of the solution was not distributed over the fiber.

Dawson infers from his results that drying with sand for 4 hours with stirring gives approximately accurate results, though possibly an additional 2 hours would be better, and that drying with asbestos for 4 hours gives results about 0.5 per cent higher than true results. He hopes to continue his investigation.

Van Zoeren, interpreting the term "to constant weight" literally, endeavored to realize an absolute constancy, or, at least, one not exceeding a change of 0.01 per cent of the original weight per hour. Finding in his experiments on Sirup No. 1 at 100°C. that such constancy was not obtainable within less than 40 hours at 100°C., he made his first weighings on the other sirups after 30 hours, his second after 40 hours, and his third after 50 hours. These experiments were made with a small electrically heated oven of Sargent's make, an oven exhibiting much greater variations of temperature than the Freas oven. Aluminium dishes 7.5 cm. in diameter and 1.8 cm. in depth were used in some cases, and dishes of 6.0 cm.  $\times$  1.5 cm. in others. The sand was washed with hydrochloric acid and ignited. The asbestos was tremolite, such as is ordinarily used in Gooch crucibles. In all cases, 5 grams of sirup were weighed in a sugar dish and transferred to the tared dish with a small



quantity of distilled water. The results at 40 hours are given in Table 5. With one exception, they show lower percentages of dry matter than were obtained by the refractometer but, considering the long period of heating, the differences are remarkably small.

TABLE 5.

*Determination of dry matter in sirups.*

(Analyst, G. J. Van Zoeren.)

SAMPLE NUMBER	DRIED 40 HOURS AT 100°C. UNDER ATMOSPHERIC PRESSURE. SPREADING MATERIAL			DRIED 100 HOURS AT 70°C. UNDER REDUCED PRESSURE. SPREADING MATERIAL			DRY MATTER BY REFRACTO- METER
	Tremolite asbestos	Sand	Pumice stone	Asbestos	Sand	Pumice stone	
	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>	<i>per cent</i>
1.....	66.84	66.62	67.69	67.42	67.36	68.29	67.37
	66.79	66.69	67.76	67.78	67.34	68.24	.....
Average.....	66.81	66.66	67.73	67.60	67.35	68.27	67.37
2.....	64.89	65.18	64.80	64.90	65.19	66.12	65.04
	64.98	64.71	64.69	64.87	64.76	64.32	.....
Average.....	64.94	64.95	64.75	64.89	64.98	65.22	65.04
3.....	67.05	67.01	66.80	67.32	66.99	67.82	67.34
	67.25	67.05	67.52	67.95	67.11	67.46	.....
Average.....	67.15	67.03	67.16	67.64	67.05	67.64	67.34

TABLE 6.

*Mean variations of individual results by drying from refractometer results.*

SAMPLE NUMBER	AT 100°	AT 70°	SPREADING MATERIAL	AT 100°	AT 70°
1.....	±0.54	±0.38	Asbestos	-0.28	±0.23
2.....	±0.21	±0.42	Sand	±0.43	±0.18
3.....	±0.29	±0.30	Pumice	±0.34	±0.70

The drying experiments conducted by the writer on sirups at 100°C. were confined to a single series in which a reboiled and cotton-wool filtered sample of Sirup No. 1 was dried on the three spreading materials and samples of Sirup No. 2, also reboiled and refiltered, were dried on asbestos. All the portions were dried at once in the same Sargent oven that Van Zoeren used, and the weight of sample and method of weighing were the same as his. For Sirup No. 2 and for the second of each pair of duplicates on Sirup No. 1, 7.5 cm. aluminium dishes were used. In all cases, the drying was more rapid in the 7.5 cm. than in the 6 cm.

dish. Weighings were made after 5, 7, 8, 9, 10, 11, 14 and 16 hours. In the final 2 hours of drying the loss of weight per hour was less than 0.1 per cent in all the 7.5 cm. dishes and in all 6 cm. dishes except the one in which Sirup No. 1 was dried on asbestos. In that dish and the two pumice dishes, the percentages of residue at the end of the 16 hours were decidedly higher than those deduced from the refractometer observations. The other asbestos portion and the two portions dried on sand gave results according closely with the refractometric indication (Table 7). On the other hand, the two sirups prepared from Sample No. 2 and dried on asbestos gave results for dry matter which, even at the end of 5 hours' drying, were lower than those derived from the refractometer readings. Considering the inadequate control of conditions, the discordance of the results obtained, and the fact that the sirups used were not the original collaborative samples, it does not appear worth while to burden the report with the details.

TABLE 7.

*Determination of dry matter in a prepared sample of Sirup No. 1 (16 hours at 100°C. under atmospheric pressure).*

(Analyst, J. F. Snell.)

MEDIUM	SIZE OF DISH	RESIDUE
	cm.	per cent
Asbestos.....	6.0	67.21
Asbestos.....	7.5	66.96
Pumice.....	6.0	67.56
Pumice.....	7.5	67.30
Sand.....	.0	66.70
Sand.....	7.5	66.62
By refractometer.....	...	66.79

One point, however, must be mentioned. Among the fifty weighings made after the ninth hour of drying no less than twelve showed an increase of weight, instead of a decrease. As the dishes were always covered during the weighings and as other dishes cooled in the same desiccator showed no similar increase, and, furthermore, as similar results were later obtained with the sugars, the only conclusion to be reached is that simultaneously with the elimination of water some chemical change (possibly an oxidation) resulting in an increase of weight is taking place. This is a question that deserves further study.

#### EXPERIMENTS AT 70°C.

The experiments at 70°C. were carried out in the Freas oven, a vacuum desiccator connected with a filter pump through a large safety-bottle being used as the vacuum chamber. In the experiments conducted by

the writer, a slow current of air, dried by passing through sulphuric acid, was allowed to flow through the chamber, and the pressures, as measured by a mercury manometer connected between the safety bottle and the vacuum chamber, were from 60 to 160 mm. Van Zoeren used the same oven and pump but a larger desiccator and admitted no current of air. He did not measure the pressures.

Van Zoeren's results at the end of 160 hours are given in Table 5. The average results are closer to the refractometer indications than are those obtained by drying 40 hours at 100°C. But when the individual results are examined, as is done in Table 6, there is but little choice between the two methods.

The only experiment conducted by the writer with sirup at 70°C. was made with a reboiled and cotton-wool filtered portion of Sample No. 3. This gave 66.04 per cent of total solids by the refractometer, and the residues after 18 hours' heating (which did not result in absolute constancy) were 66.18 and 66.32 per cent in two 7.5 cm. dishes, and 66.40 per cent in a 6 cm. dish.

With sugars, constant weight was realized by both experimenters in about 48 hours but results were not concordant. These results, as well as those obtained with the sugars at 100°C., are omitted on account of such discordance.

#### WINTON LEAD NUMBER.

The points indicated for study in reference to the Winton method were:

(1) The advisability of substituting 25 grams of cane sugar sirup for the few drops of acetic acid in the blank determination<sup>1</sup>.

(2) The advisability of reducing the results to the dry matter basis<sup>2</sup>.

The results of the collaborative work are shown in Table 8. The basic acetate solution was prepared from Horne's salt. In the experiments conducted by the writer the acetic acid blank was treated with a few drops of 2N acetic acid. On diluting, slight clouding ensued, but the weights of lead sulphate obtained by precipitation after settling were practically identical with those obtained from the cane sugar blanks, which remained quite clear. Van Zoeren, who added enough acetic acid to prevent precipitation, obtained 2.2 mg. less lead sulphate from these blanks than from the cane sugar blanks.

The results may be interpreted as slightly favorable to the use of the cane sugar sirup. This has also a logical advantage over acetic acid, in that the substance added is identical with that which, in the case of the

<sup>1</sup> *J. Ind. Eng. Chem.*, 1913, 5: 997.

<sup>2</sup> *Ibid.*, 1914, 6: 221.



TABLE 8.

*Collaborative results on Winton lead number.*

SAMPLE NUMBER	ACETIC ACID BLANK		CANE SUGAR BLANK	
	Van Zoeren	Snell	Van Zoeren	Snell
1.....	1.82 1.84 1.83 .... ....	About 0.01 per cent	1.92 1.94 1.93 .... ....	1.89 1.89 1.88 1.88 1.89
4.....	1.73 1.81 1.77		1.79 1.91 1.85	2.07 2.08 2.08
2.....	2.90 2.82 2.86 .... ....	lower than with the cane sugar blank	2.99 2.91 2.95 .... ....	2.29 2.31 2.47 2.47 2.39
5.....	2.23 2.22 2.23		2.33 2.30 2.31	2.50 2.47 2.49
3.....	2.36 2.38 2.37	..... ..... .....	2.45 2.48 2.47	2.43 2.40 2.42
6.....	2.41 2.38 2.39	..... ..... .....	2.56 2.47 2.52	2.39 2.36 2.38

maple sirup, prevents the precipitation of the basic acetate. If it were adopted, the directions would be to make a blank determination, using 25 cc. of a pure cane sugar sirup (sp. gr. 1.320) in place of the maple sirup.

The question of the advisability of abandoning the reduction of Winton lead numbers to the dry basis is one which does not permit of solution upon the basis of work on a few samples. The reduction to dry basis was not a part of the original method of Winton and Kreider<sup>1</sup> and, as has been shown by Snell and Scott<sup>2</sup>, the range of variation of wet basis Winton numbers in genuine maple sirups is narrower than that of the dry basis numbers. The wet basis number is therefore a sharper criterion for the detection of adulteration than the dry basis.

Sherwood favors the retention of the reduction to dry basis on the grounds that the difference of range is not great; analyses of maple products published by the Bureau of Chemistry and certain other investigators and frequently used as bases of comparison are stated on the

<sup>1</sup> *J. Am. Chem. Soc.*, 1906, 28: 1204.<sup>2</sup> *J. Ind. Eng. Chem.*, 1913, 5: 997.

dry basis, and to discontinue the calculation of the Winton number to dry basis would tend to confusion. Opinions have not been expressed by other collaborators.

The directions of the Winton lead method should be closely followed to insure correct results. The sugar solution must not be warm nor can it be poured into the lead subacetate. The following parallel results on Sample No. 5 illustrate this point:

TABLE 9.  
*Determination of lead number on Sample No. 5.*

DESCRIPTION OF METHOD	LEAD NUMBER
Regular method. ....	2.49
25 cc. of subacetate solution, measured into a flask, sirup added and washed in with water. ....	2.69
The diluted sirup heated to 60°C. and the subacetate added. ....	2.26

#### CANADIAN LEAD NUMBER.

The Laboratory of the Canadian Inland Revenue Department has for many years used this value as the chief criterion for the discrimination of genuine and adulterated maple products. The directions for its determination, as approved by A. Valin of that laboratory, are as follows:

Weigh the quantity of sirup containing 25 grams of dry matter, transfer to a beaker, add 50-75 cc. of water, boil gently for 2-3 minutes, transfer to a 100 cc. flask, cool and make up to the mark.

Pipette 20 cc. of this solution into a large test tube, add 2 cc. of lead subacetate solution (sp. gr. 1.26) and mix. Allow to stand 2 hours, filter through a tared Gooch, wash four or five times with boiling water, dry at 100°C. and weigh. Multiply the weight of the dry precipitate by 20.

The points indicated for study were:

- (1) The advisability of weighing 25 grams of sirup instead of the quantity of sirup containing 25 grams of dry matter.
- (2) The necessity for boiling the diluted sirup if all samples have been boiled in the preparation for analysis.
- (3) The advisability of substituting 1.25 for 1.26 as the density of the subacetate solution.
- (4) The advisability of defining the volume of wash water and allowing more license as to temperature<sup>1</sup>.
- (5) Variations between duplicates.

The collaborative results are given in Table 10. Van Zocren and the writer used a subacetate solution (sp. gr. 1.25) prepared from Horne's

<sup>1</sup> *J. Ind. Eng. Chem.*, 1913, 5: 996.

salt. They also used a 100 cc. beaker in place of the large test tube. The subacetate solution was added from a burette. The writer washed the solution as directed without close attention to the quantity of wash water. Van Zoeren always washed the solution with exactly 100 cc. of boiling water. It should be noted that the mat of asbestos in the Gooch crucible in this determination needs to be heavier than for most other precipitates. Inattention to this detail and to the exact volume of the wash water may possibly account for the inferior agreement of the writer's duplicates as compared with those of the other collaborators.

TABLE 10.  
*Collaborative results on Canadian lead number.*

SAMPLE NUMBER	COLLABORATORS			USING 25 GRAMS SIRUP AND CALCULATING TO DRYNESS
	Valin	Van Zoeren	Snell	Van Zoeren
1.....	2.20	2.51	2.55	1.96
	2.24	2.57	2.04	1.93
	2.20	2.49	2.28	1.74
	2.18	....	....	1.68
Average.....	2.20	2.52	2.29	1.83
4.....	2.22	2.52	2.24	1.69
	2.22	2.48	2.23	1.72
	2.08	....	....	....
	2.22	....	....	....
Average.....	2.19	2.50	2.24	1.71
2.....	3.22	4.04	4.30*	3.76
	3.24	4.01	3.94	3.56
	3.16	4.07	3.59	3.40
	3.16	3.92	3.73	3.46
Average.....	3.19	4.01	3.75	3.54
5.....	2.84	3.12	3.12	2.83
	2.74	3.15	3.35	....
	2.80	....	....	....
	2.70	....	....	....
Average.....	2.77	3.14	3.24	2.83
3.....	3.22	3.31	3.52	3.16
	3.06	3.36	3.42	3.08
	3.10	3.32	....	3.18
	3.20	3.32	....	3.16
Average.....	3.14	3.33	3.47	3.15
6.....	3.16	3.32	3.33	3.07
	3.14	3.29	3.23	3.17
	3.08	....	....	....
	3.06	....	....	....
Average.....	3.11	3.31	3.28	3.12

\* Not included in average.

Valin admits that it would be more convenient to weigh 25 grams of sirup instead of 25 grams of dry matter, but states that the Laboratory of the Canadian Inland Revenue Department follows the other procedure in order to have a uniform standard for sirup and sugar. In the process of this association in which the sugar is converted into sirup for analysis, this object is attained in another way, and the only objection to adopting the procedure with 25 grams of sirup is that the results so obtained would not, even after reduction to a dry basis, be comparable with those which the Laboratory of the Canadian Inland Revenue Department now publishes. On the other hand, they would be comparable with the results which that laboratory has published upon a large number of genuine sirups<sup>1</sup>. This is not made clear in the publication itself, but in answer to an inquiry, the Chief Analyst advised the associate referee under date of February 17, 1913, that the lead determinations were made on 5 grams of the sirup, and the results were later calculated to a dry basis.

The wide difference which usually exists between dry basis results obtained with the use of a definite weight of sirup and those obtained with the quantity of sirup containing that definite weight of dry matter has been pointed out by Snell and Scott<sup>2</sup> and is further illustrated by results obtained by Van Zoeren on the present collaborative samples, particularly on Samples 1 and 4 (Table 10). In all cases, his results, using 25 grams of sirup, are lower than those using 25 grams of dry substance. This is consistent with the work of Snell and Scott and is what one would expect in view of the solvent action of sugar upon the precipitate. It is notable, however, that in four instances out of six, the results with 25 grams of sirup are equal to or greater than Valin's results obtained with the use of 25 grams of dry substance. This is doubtless only a detail of the more general fact that Valin's results as a whole are lower than those of the other collaborators. Whether this is due to a difference in the lead subacetate solutions used, or to some variation in the procedure, is not known.

The only results bearing on the question of the necessity of boiling the diluted sirups when they have been previously boiled in the preparation of the sample are those of the associate referee on Sirup No. 2. A portion of this sirup, prepared for analysis by boiling to 104°C. and filtering through cotton wool, gave 3.60 and 3.50 as the Canadian lead number without reboiling, and 3.22 and 3.44 when the sirup was reboiled after dilution.

Valin is of the opinion that the use of subacetate solution of specific gravity of 1.25 instead of 1.26 would make no difference in the results.

<sup>1</sup> Can. Lab. Inland Rev. Dept., Bull. 228: (1911).

<sup>2</sup> J. Ind. Eng. Chem., 1913, 5: 997.

Since the former strength is now commonly used in clarifying for the polariscope, its use is to be recommended on the score of convenience.

Further study on the Canadian lead method is strongly recommended since this method is so simple and possesses the advantage that in adulterating with refined sugar the values fall off more rapidly than the percentage of maple sirup<sup>1</sup>.

#### ASH VALUES.

Van Zoeren and the writer have done considerable work on the ash values but as no results from collaborators were received our results are reserved for later publication.

#### OTHER METHODS.

The electrical conductivity method<sup>2</sup> should be further tested before it receives official recognition. It is a very simple, rapid test. In the writer's laboratory, it has given results of a more restricted range in genuine sirups than any of the recognized methods. For collaborative purposes, it might perhaps be well to dilute the sirups to a more definite sugar content than is directed for the rapid test, but whether the increase of accuracy would compensate for the loss of time is a point that would require study.

The volumetric lead method<sup>3</sup> has not given satisfactory results with the present samples. With sirups of high quality, there is sometimes room for difference of opinion as to the plotting of the graphs.

#### RECOMMENDATIONS.

It is recommended—

(1) That work on the preparation of the sample be continued with a view to revision of the directions.

(2) That collaborative work be done on the Winton lead number.

(3) That the Canadian lead number and conductivity value methods be further studied collaboratively with a view to their adoption.

(4) That work on the determination of moisture and ash be resumed when these topics are under study in reference to other saccharine products.

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<sup>1</sup> *J. Ind. Eng. Chem.*, 1913, 5: 995.

<sup>2</sup> *Ibid.*, 1916, 8: 331.

<sup>3</sup> *Ibid.*, 1916, 8: 241.